A LOW TEMPERATURE AND PRESSURE METHANOL SYNTHESIS

MUHAMMAD ARIF BIN ZAINAL

CHEMICAL ENGINEERING
UNIVERSITI TEKNOLOGI PETRONAS
JULY 2008

A Low Temperature And Pressure Methanol Synthesis

by

Muhammad Arif bin Zainal

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

JULY 2009

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

A Low Temperature and Pressure Methanol Synthesis

by

Muhammad Arif bin Zainal

A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirements for the
BACHELOR OF ENGINEERING (HONS)
(CHEMICAL ENGINEERING)

Approved by,

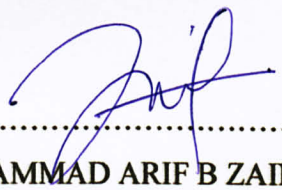
.....

(Dr. Chandra Mohan)
Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
July 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



.....

(MUHAMMAD ARIF B ZAINAL)

ABSTRACT

Researchers and industrial practitioners have been discussed a lot about methanol synthesis nowadays. This is because due to the high demand of methanol itself in processing formaldehyde, chloromethanes, amines, acetic acid, MTBE and methyl methacrylate. In addition of that, currently methanol synthesis using syngas has attracted this researchers and industrial practitioner since it involving greenhouse gas which is CO_2.

Methanol synthesis reaction is an exothermic reaction. Researchers are still trying to find a way to lower down the operating conditions (e.g temperature and pressure) because of controlling the temperature inside the reactor is difficult. Besides, high operating conditions may deactivate the catalyst, thus lowering down the CO conversion, yield and not less may lowering down the selectivity of methanol. Since that sulphur free synthesis gas has been able to produce, Cu-based catalyst, a more active catalyst can be used and a low pressure and temperature methanol synthesis is more feasible.

The study on low temperature methanol synthesis using 2-butanol has been successful and currently been applied in big scale. However, there is still no well-establish study on low temperature and pressure using aromatics solvent as catalytic solvent, thus gain the author interest to study on this matter.

In the first semester, the author come out with some theory about how methanol is produced after done some deep research. The project continued in the second semester with catalyst preparing, catalyst characterization, run methanol synthesis reaction in a three phase reactor and samples analysis. In this project, two aromatics solvents (toluene and xylene) is use in the process of synthesizing methanol at temperature of 150 - 200°C and pressure of 20 bar. Percentage of total carbon conversion and yield will be observed as well as the selectivity at the end of this project.

ACKNOWLEDGEMENT

First and foremost, the author would like to praise Allah the Almighty for with His blessings and guidance the author is able to successfully complete my Final Year Project in partial fulfillment of the requirements for the bachelor of engineering (hons).

This project, conducted in the Chemical Engineering Department of Universiti Teknologi PETRONAS, was done with the help and support of many people whom I would like to thank.

Firstly, the appreciation goes to my Final Year Project supervisor, Dr Chandra Mohan. His continuous motivation and commitment to this project has made it more meaningful and worthwhile. While maintaining his professionalism as a lecturer and supervisor, he also made effort to understand the difficulties and problem which I encountered.

Heartfelt appreciation is given to University Technology PETRONAS (UTP) and Chemical Engineering Department for equipping students with essential skills and excellent theoretical as well as practical work. In conducting this study, a lot of manpower was required. The experiment that I've done for this project could not be done successfully without the help and expertise of the laboratory technician of Mechanical, Chemical and Electrical & Electronic Engineering Department who have been very helpful, unconditionally.

Undertaking this project has been a great challenge, physically and mentally. My friends have been there to provide support and help. I would like to personally convey my appreciation to fellow colleagues, who have contributed directly or indirectly to the completion of this project.

Last but not least, my deepest appreciation to my father, mother, brother and sister for their never-ending, care and patience. I could not have done this without their encouragement and understanding.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 PROJECT BACKGROUND

Methanol is the simplest alcohol compound, comprised of one carbon atom, one oxygen atom and four hydrogen atoms. Methanol is a monohydric alcohol. It melts at -97.8°C and boils at 67°C. Nowadays, methanol also can be referred to wood alcohol, carbinol and metyl alcohol. Methanol is poisonous, flammable and relatively volatile. It has no taste or color, but it does have a slight scent. Small internal doses, continued inhalation of the vapor, or prolonged exposure of the skin to the liquid may cause blindness. As a result, commercial use of methanol has sometimes been prohibited. Methanol is very commonly used as a feedstock in the chemical industries. It is also used as fuel, as antifreeze and as solvent. Methanol is used when producing for example formaldehyde, acetic acid and methyl tertiary butyl ether (MBTE).

The commercial methanol synthesis was implemented in 1923 by BASF in Germany. It is now produced synthetically by the direct combination of hydrogen and carbon monoxide gases, heated under pressure in the presence of a catalyst. This process was known as the "high-pressure" process.

1.2 PROBLEM STATEMENT

The first methanol synthesis was implemented in 1923 by BASF in Germany. The operation point in this process was 250 - 350 bar and 320 - 450 °C [1]. This process was known as the "high-pressure" process. The catalyst used in this process was based on chromate. In the 1960s, the so called "low-pressure" process replaced the old one. The ability to produce sulfur free synthesis gas made it possible to use the more active Cu-based catalysts. The process was operated in the lower pressure and temperature, 60

- 80 bar and 250 - 280 °C [1]. Two low-pressure processes dominate the markets nowadays: ICI and Lurgi processes [1, 2].

Methanol is commercially produced by ICI process under high temperature and pressure, using cooper-zinc based oxide catalyst and alcohol as catalytic solvent in multibed quench reactor [1, 2]. Since that methanol synthesis is an extremely exothermic reaction, this process efficiency is severely limited by thermodynamics. For example, at 573 K and 5MPa, the theoretical maximum of one-pass CO conversion is around 20% [2]. The recycle of unreacted reactant is necessary to enhance the reactant conversion. This will leading to the production cost higher. The recycling process can be omitted if one pass conversion is high enough [14]. Thus, developing a high one-pass conversion process at lower temperature which is of the thermodynamic favor will greatly lower the cost of production [14]. This low temperature and pressure methanol synthesis has gain interest from worldwide. Researchers have found ways of producing methanol in a low temperature and pressure process. Nowadays, the most active or highest potential low temperature and pressure process is using Cu/ZnO/Al2O3 catalyst and 2-butanol as catalytic solvent. So far, there is no theoretical study on methanol synthesis using Cu/ZnO/Al2O3 catalyst and aromatics as catalytic solvent. This has gain interest from the author to do the study.

1.3 OBJECTIVE AND SCOPE OF STUDY

The objectives of this project are:

- To study the effect of product selectivity on the reaction of hydrogen and carbon oxides using $Cu/ZnO/Al_2O_3$ catalyst with aromatic solvent as liquid medium in 3 phase slurry reactor.

Scope of project that covered in this project:

- Catalyst Preparation
- Catalyst Characterization
- Methanol Synthesis
- Liquid and Gas Samples Analysis

CHAPTER 2
LITERATURE REVIEW

2.1 METHANOL

Methanol (CH_3OH), also known as wood alcohol, is considered an alternative fuel under the Energy Policy Act of 1992. Today, most of the world's methanol is produced by a process using natural gas as a feedstock.. As an engine fuel, methanol has similar chemical and physical characteristics as ethanol [15]. Methanol is methane with one hydrogen molecule replaced by a hydroxyl radical (OH). It is predominantly produced by steam reforming natural gas to create a synthesis gas, which is then fed into a reactor vessel in the presence of a catalyst. This process then produces methanol and water vapor.

Although a variety of feedstocks can be used to create methanol, today's economics favor the use of natural gas [15]. Methanol is a colourless liquid that may explode when exposed to an open flame. Methanol has a flash point of 12°C and a boiling point of 65°C. It occurs naturally in wood and in volcanic gases. Methanol is also a product of decaying organic material. It is manufactured from natural gas in large quantities (approximately 28,000,000 tonnes in 2001) [3]. The largest users of the methanol sold in the United States are companies that make methyl t-butyl ether (MTBE), an oxygenate that is blended with gasoline to enhance octane and create cleaner burning fuel.

MTBE production and use has declined in recent years because it has been found to contaminate ground water. This use is likely to reduce in the USA, however, the MTBE demand is increasing in Europe. Companies also use methanol to make chemicals such as formaldehyde, acetic acid, chloromethane, and methyl methacrylate[3]. **Table 1** below shows the application of methanol nowadays [4].

Product (in w.%)	1988	1996	2005
Formaldehyde	39	35	38
Acetic acid	6	7	11
Methyl halides	7	7	7
MTBE	12	27	20
Dimethyl terephthalate	3	2	2
Methyl amines	4	4	4
Methyl methacrylate	3	3	4
Solvents	9	4	4
Others	17	11	13
Sum (in mio t/a)	17,3	24,3	32,1

Figure 1: Application of Methanol

2.2 METHANOL SYNTHESIS

2.2.1 Reaction Chemistry and Thermodynamics

Syngas is a mixture of CO, CO2 and H2 produced from various sources through steam reforming, partial oxidation, CO2 reforming or auto thermal reforming [1]. In methanol synthesis, either CO or CO2 or both hydrogenates to methanol. The reactions are:

$$CO_2 + 3H_2 = CH_3OH + H_2O \qquad (\Delta H = -50 \text{ kJ/mol})$$

$$CO + H_2O = H_2 + CO_2 \qquad (\Delta H = -41 \text{ kJ/mol})$$

Thus, giving a net reaction of:

$$CO + 2H_2 = CH_3OH \qquad (\Delta H = -91 \text{ kJ/mol})$$

The production of methanol is strongly influenced by thermodynamics. The thermodynamic equilibrium limits the process to a low conversion and thus the recycling of the outlet is required if a high conversion is desired. The overall reaction is also strongly exothermic and thus a significant cooling is required [1].

2.2.2 Mechanism Of Methanol Formation

Figure 1 below shows the mechanism of methanol formation on $Cu/ZnO/Al_2O_3$ [5]:

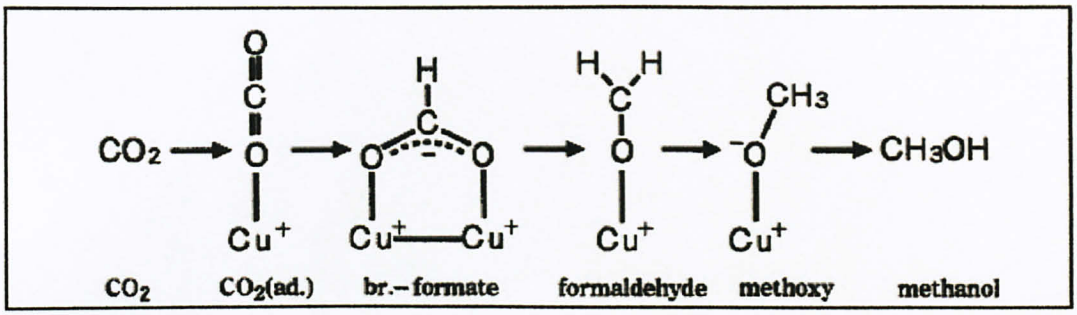


Figure 2: Methanol Formation

CO_2 is linearly absorbed on the Cu^+ site. The H atom, which is probably absorbed on the metallic cooper, attacks the C atom of the adsorbed CO_2 and a formate intermediate is formed. Then, H atoms attack the C and the O atoms of the formate species, and the C-O bond is broken simultaneously. A formaldehyde-type intermediate is formed on the Cu^+ site. The H^- on Zn, which is adsorbed on ZnO as a result of the heterogeneous dissociation of H_2, attacks the C atom of the formaldehyde-type species, and a methoxy intermediate is formed. Furthermore, the H+ on the O atom of ZnO attacks the O atom of the methoxide, and finally, methanol is produced [5].

2.2.3 Comparison on Paper About Methanol Synthesis

Table 1: Comparisons on Methanol Synthesis Paper

	Title of Papers			
Characteristic	Development of a new low-temperature methanol synthesis process	A new method of low-temperature methanol synthesis on $Cu/ZnO/Al_2O_3$ catalyst from $CO/CO_2/H_2$	Catalytically active supercritical fluid to accelerate methanol synthesis Catalytically active supercritical fluid to accelerate methanol synthesis	
Reactor Operating Condition	Flow type Semi batch	Continuous Reactor	Converted fixed-bed	Converted fixed-bed
Pressure (bar)	50	30	55	30.4
Temperature range (K)	273-443	443	538	507.6
Speed of stirrer (rpm)	1260	2000	-	-
Catalyst				
Catalyst Preparation	Co-Precipitation	Impregnation Method		
Catalyst Used	Cu/ZnO (3.0g)	$Cu/ZnO/Al_2O_3$	Cu/ZnO based of ICI Co. (0.50g)	Cu/ZnO based of ICI Co. (0.50g)
Solvents	Different type of alcohols	Ethanol	2-Butanol	n-hexane
Results	Solvents use and results for the reaction are: • Solvents; o 1 – Ethanol o 2 – 1-Propanol o 3 - 2-Propanol o 4 – 1-Butanol o 5 – 2-Butanol o 6 – *Iso*-Butanol o 7 – 1-Pentanol o 8 – 2-Pentanol o 9 – 1-Hexanol	Result gathered using uncalcined catalyst with Cu diameter 22nm and reduction Temperature at 493K. Molar ration of Zn/Cu is 1/1 shows the		

	○ • Results;									highest conversion and selectivity		
	1	2	3	4	5	6	7	8	9			
Total carbon conversion (%)	33.5	35.2	44.4	34.4	47.0	29.9	34.2	44.0	34.5	14.4	48.1	21.4
Selectivity (%)	33.0	34.5	44.0	34.3	46.5	29.8	34.1	43.3	34.4	96.4	90.7	98.1
Byproduct	Methyl Formate & Ester (HCOOR)									Ester (HCOOR)	Ester (HCOOR)	CH_4

2.3 CATALYST

Catalysts increase the reaction rates of the chemical reactions but are not consumed in the reactions. They lower the energy required for the initiation of the reactions and thus make the reactions easier to occur. Catalysts do not have influence on the position of the equilibrium and do not enable the reactions that are forbidden by the thermodynamics [1]. Typically more than one chemical reaction occurs in chemical reactors. Catalysts may influence only the desired reaction and thus increase the selectivity of the process. The structure of the catalyst is important concerning its usability. High activity and selectivity are ensured if the catalyst has the correct chemical components and a high enough surface area [1].

The first catalyst used for the methanol production from the synthesis gas was ZnO/Cr2O3 [1].It was commercialized by BASF and used in the high pressure and high temperature synthesis [1]. The improvements in the purification of the methanol synthesis feed (especially removal of sulphur) led to a major improvement in catalysts as the use of Cu-ZnO catalyst was enabled [1]. The Cu/ZnO catalyst has a high activity and the synthesis can be operated at the lower pressure and temperature. Nowadays, the $Cu/ZnO/Al_2O_3$ catalyst is almost exclusively used in industry [1].

Since $Cu/ZnO/Al_2O_3$ has the highest activity toward syngas, this catalyst has been chose in this project.

2.4 PARR HIGH PRESSURE REACTOR

Below in **Figure 3** is the exact reactor that will be used in the project.

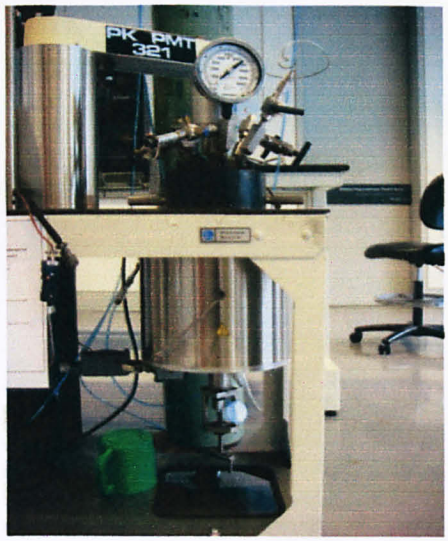


Figure 3: PARR High Pressure Reactor

2.4.1 Pressure and Temperature Limits

Table 2: Pressure and Temperature Limits

Pressure Limit	Temperature Limit
1450 psig 145 bar	500 °C

2.4.2 Valves

Gas Inlet Valve

The gas inlet valve is connected to a dip tube which extends to a point near bottom of the cylinder. This valve can be easily identified when the bomb is open.

Gas Release Valve

The gas release valve is installed in a port without any attachments installed on the underside of the head. Gas released from this valve will be drawn from the headspace of the vessel.

Liquid Sampling Valve

The liquid sampling valve is attached to the same fitting as the gas inlet valve and connected to the same dip tube. With this arrangement, incoming gas is always introduced below the surface of the liquid and the operator is provided with a means for clearing the dip tube to be sure that any sample taken during a run will be representative of the charge. This can be done by opening the upper gas inlet valve momentarily to allow the inlet gas to force any liquid in the dip tube back into the reactor before withdrawing a sample from the sampling valve.

Pressure Gauge

A pressure gage, usually 0-5000 psi with a T316 stainless steel Bourdon tube [16], is mounted to the head with a coned adapter fitting. Gages are furnished in a variety of ranges to meet individual needs.

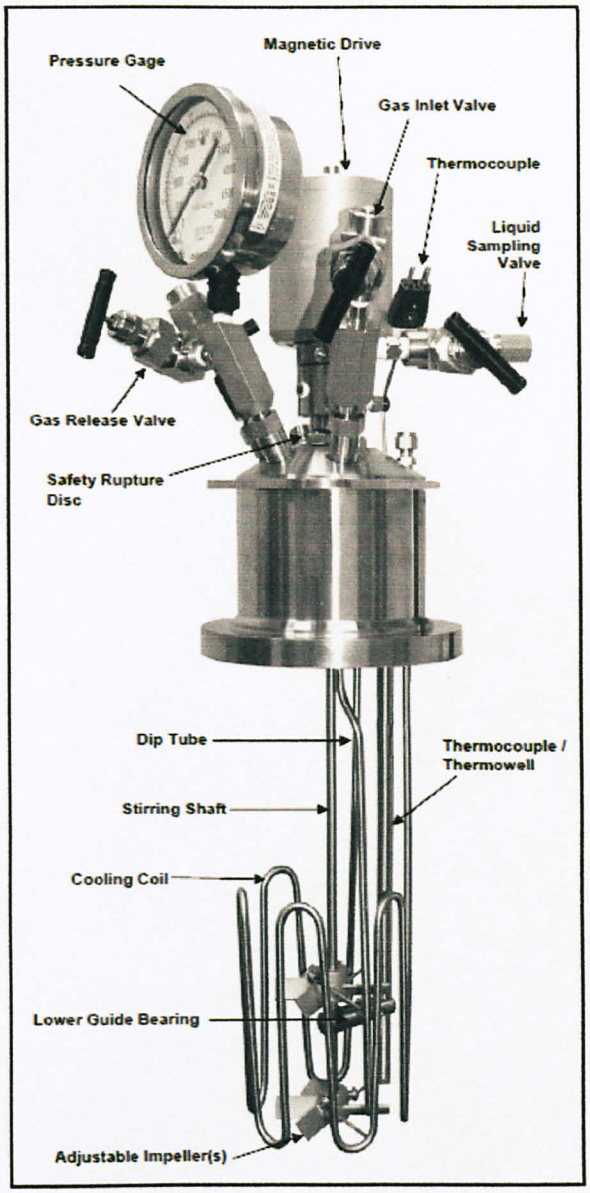


Figure 4: The Valves

2.4.3 Slurry Three Phase Reactor

Slurry three-phase reactor has three phases in which solid catalyst particles are held in suspension in a liquid phase and contacting with gas phase reactant.

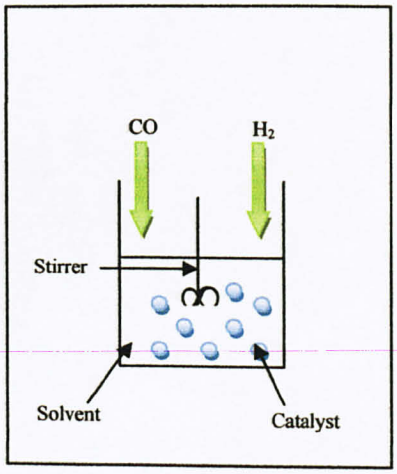


Figure 5: Illustrative picture inside the reactor

Conventional methanol reactors use fixed beds of catalyst pellets and operate in the phase [6]. The slurry three phase reactor uses catalyst in powder form, and slurried in aromatic solvents which are toluene and xylene. The solvents act as a temperature moderator and a heat removal medium, transferring the heat of reaction from the catalyst surface via the liquid slurry to water [6]. The stirrer functions are to stir the solvent with catalyst to contact with the gas reactant at the solvent's surface and to uniformly divide heat in the solvent.

2.5 CATALYST CHARACTERIZATION

2.5.1 Temperature Programmed Desorption (TPD)

TPD analysis allows defining the strength, the number and the type of active sites available on the surface of a catalyst by the determination of the quantity of gas desorbed from the catalyst submitted to a linear temperature ramp.

2.5.2 Temperature Programmed Reduction (TPR)

TPR analysis allows determining the number and quantity of the reducible species present in the sample and the temperature at which the reduction itself takes place as a function of the flow conditions, the percentage of reactive gas, the quantity of sample and the speed of the temperature increase. The gas used for this type of analysis is a mixture of reactive gas with an inert gas, as hydrogen in argon or nitrogen at 5 or 10%.

2.5.3 Temperature Programmed Oxidation (TPO)

The TPO analysis allows evaluating the temperature range, in which a sample undergoes oxidation due to an oxidizing agent contained in a gas mixture such as O2/He 5% that is flown inside the reactor containing the sample.

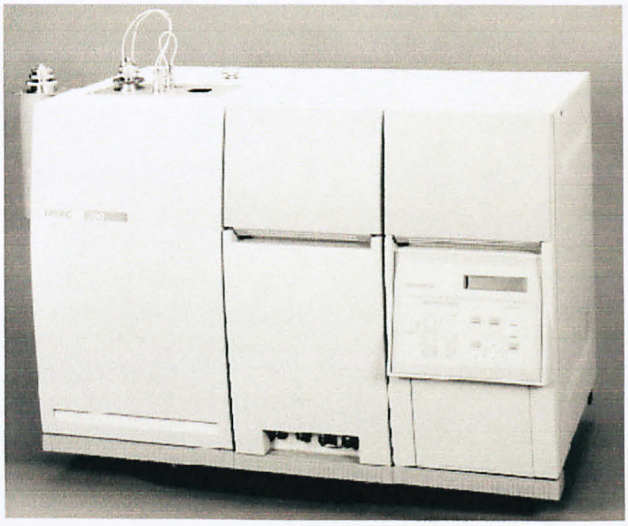


Figure 6: TPDRO 1100

2.5.4 Other Catalyst Characterization

Table 3: Other Catalyst Characterization

No.	Characterization Test	Function/Objective
1.	AAS (Atomic Absorption Spectroscopy)	• Provides accurate *quantitative analysis* (determining the concentration) for metals in water, sediments, soils or rocks. (Samples are analyzed in solution form, so solid samples must be leached or dissolved prior to analysis.) • Metal ions in a solution are converted to atomic state by means of a flame.
2	SEM (Scanning Electron Microscopy)	• Producing high-resolution images of a sample surface using electrons instead of light waves. • The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's *surface topography, composition* and other properties such as electrical conductivity. • Useful for *judging the surface structure* of the sample.
3.	TGA (Thermo Gravimetric Analysis)	• Determine *changes in weight in relation to change in temperature* • It can be used to determine characteristics of materials such as *degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues.*
5.	XRD (X-Ray Diffraction)	• Non-destructive technique that reveals detailed information about the *chemical composition*

		and crystallographic structure of materials. • Useful for *determining the arrangement of the atoms.* • *Yields information about the structure of the crystal.*
6.	BET (Brunauer, Emmett, And Teller Surface Area And Pore Size Analyzer)	• Determining the *surface area* of powders as well as the *pore size distribution* of porous materials • It is an *extension of the Langmuir theory, which is a theory for monolayer molecular adsorption to multilayer adsorption.*
7.	Temperature Programmed Desorption (TPD)	• TPD analysis allows *defining the strength, the number and the type of active sites available on the surface of a catalyst* by the *determination of the quantity of gas desorbed from the catalyst submitted to a linear temperature ramp.*
8.	Temperature Programmed Reduction (TPR)	• TPR analysis allows determining the number and quantity of the reducible species present in the sample and the temperature at which the reduction itself takes place as a function of the flow conditions, the percentage of reactive gas, the quantity of sample and the speed of the temperature increase. • The gas used for this type of analysis is a mixture of reactive gas with an inert gas, as hydrogen in argon or nitrogen at 5 or 10%.

CHAPTER 3
METHODOLOGY

3.1 CATALYST PREPARATION

Catalyst: $Cu/ZnO/Al_2O_3$

1. 139.075g of copper oxide, 89.67g of zinc acetate and 18.75g of aluminum nitrate are dissolved in 1.011 liters of water (Solution 1)
2. 158.04g of sodium bicarbonate is dissolved in 1.011 liters of water (Solution 2)
3. Both solutions are heated to 90°C separately. Solution 2 is added rapidly to solution 1. A magnetic stirrer is utilized to enhance the mixing process.
4. PH of the solution is closely monitored. The mixing is stopped when the pH of the Solution 1 is around 7.
5. The mixture is stirred for a further of 15 minutes for aging process.
6. The precipitation is then filtered off and washed nitrate-free with water.
7. The filtered catalyst is then dried in oven for overnight at temperature of 110°C in order to remove the moisture from the catalyst.
8. The dried catalyst is comminuted in order to enhance the effectiveness of the calcination process.
9. Calcination is done in furnace at temperature of 350°C for one night

3.2 SOLVENT PREPARATION

Solvent will be prepared based on weight percentage. See in **Table 1.**

Table 4: Types of Solvents

	Solution 1	Solution 2	Solution 3
Toluene	100 wt%	50 wt%	0 wt%
Xylene	0 wt%	50 wt%	100 wt%

3.3 OPERATING CONDITIONS

Operating conditions that will apply in this project is:

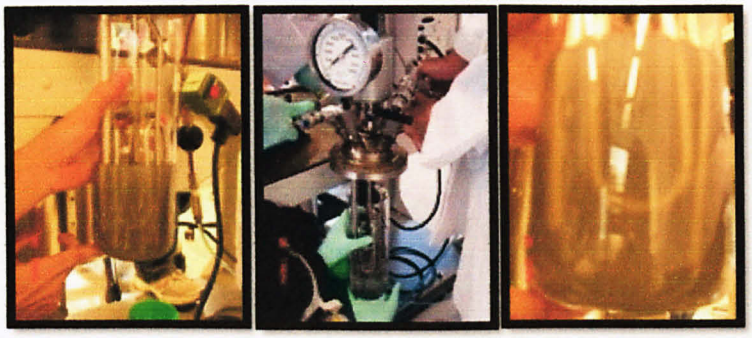
- Temperature: 150 – 200 °C
- Pressure : 30 bar
- Stirrer Speed: 300-320 ppm

3.4 SPEED CALIBRATION

1. 500ml of solvent and 2g of catalyst are prepared.
2. Both solvent and catalyst are mixed into the reactor flask.
3. Carefully insert the reactor stirrer into the reactor flask.
4. Switch ON the main power supply
5. Press the "High Limit Button" at the back of the main controller to reset the alarm.
6. Switch ON Display button.
7. Switch ON the stirrer and slowly increase the RPM of the stirrer until the catalyst is floating on the surface of the solvent. The optimum RPM gives no turbulence on the solvent's surface while maintaining the catalyst on the solvent's surface.

8. Lower the RPM of the stirrer after the suitable speed is identified and turn off the stirrer.
9. Switch OFF the display.

Figure 7: Speed Calibration



3.5 METHANOL SYNTHESIS

Prepare 500ml solvent and 2g of catalyst and mixed both of them in a container before inserting the container into the reactor.

Starting Up the Reactor

1. Switch ON the main power supply
2. Press the "High Limit Button". The High Limit will not switch off if;

    a. Pressure transducer not connected, or
    b. The controller not working.

3. Switch ON Display button
4. Wait for a few minutes until the reading on the display stabilized.
5. Insert reaction gases into the system as follow:

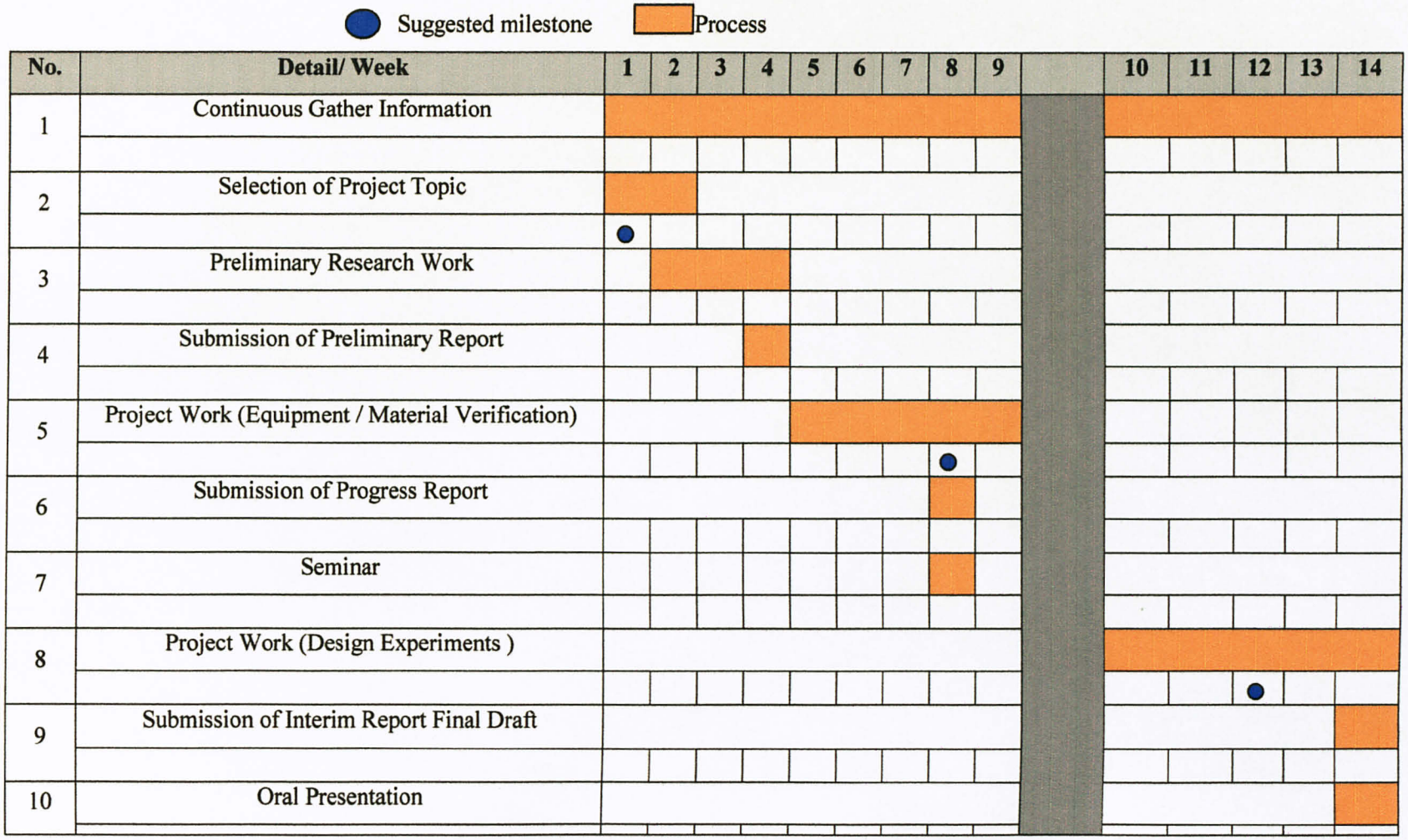
    i Open a little Carbon monoxide (CO) inlet gas valve (on top of the gas tank) while ensure the outlet valve gas (on gas regulator) is fully closed.
    ii Crack open the main gas valve that connect the reactor to the gas supply.
    iii Open a little the outlet gas valve just to ensure enough flow to the reactor.
    iv Slowly increase the outlet gas valve pressure while referring to the display until required pressure is achieved.
    v Close main gas valve when desired pressure is achieved.
    vi Close outlet gas valve follow by the inlet gas valve.
    vii Repeat the above steps for CO_2/H_2 gas until total pressure required is achieved.

6. Set temperature required for the system (150°C) and on Heater II.
7. Change from Heater II to Heater I to maintain the temperature.

8. On the stirrer after desired temperature is achieved. Set the speed of the stirrer to be 300 to 315 RPM (optimum calibrated RPM).
9. Take both gas and liquid samples at every one hour until pressure inside reactor is constant.

3.6 PROJECT MILESTONES

Below is the Gantt chart showing the project flow and milestone for the Final Year Project 1:



Below is the Gantt chart showing the project flow and milestone for the Final Year Project 2:

No.	Detail/ Week	1	2	3	4	5	6	7	8	9		10	11	12	13	14
1	Continuous Gathering Information															
2	Project Work (Conduct Project Experiment)															
				●												
3	Submission of Progress Report 1															
4	Project Work (Conduct Material Testing)															
									●							
5	Submission of Progress Report 2															
6	Seminar (compulsory)															
7	Project work (Data Analysis & Evaluate Results)															
													●			
8	Poster Exhibition															
9	Submission of Dissertation (soft bound)															
10	Oral Presentation															
11	Submission of Project Dissertation (hard bound)											7 days after oral presentation				
																●

CHAPTER 4

RESULT & DISCUSSION

4.1 SCANNING MICRON MICROSCOPY (SEM)

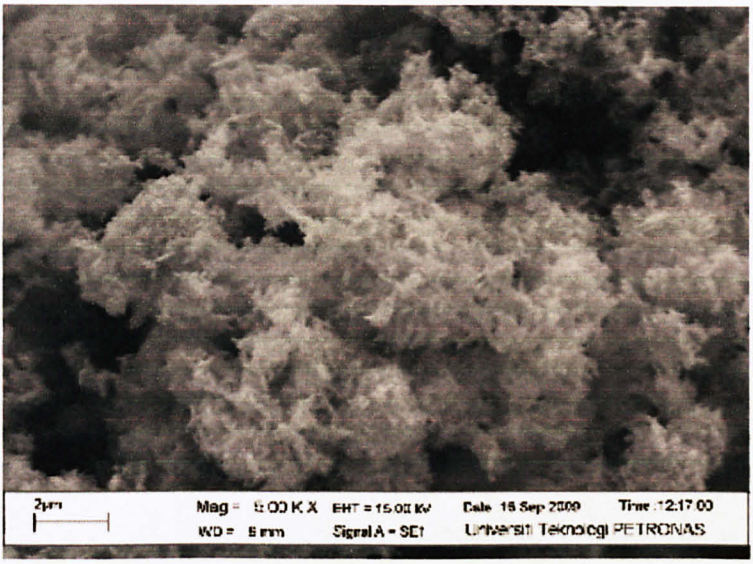


Figure 8: SEM at 5000K

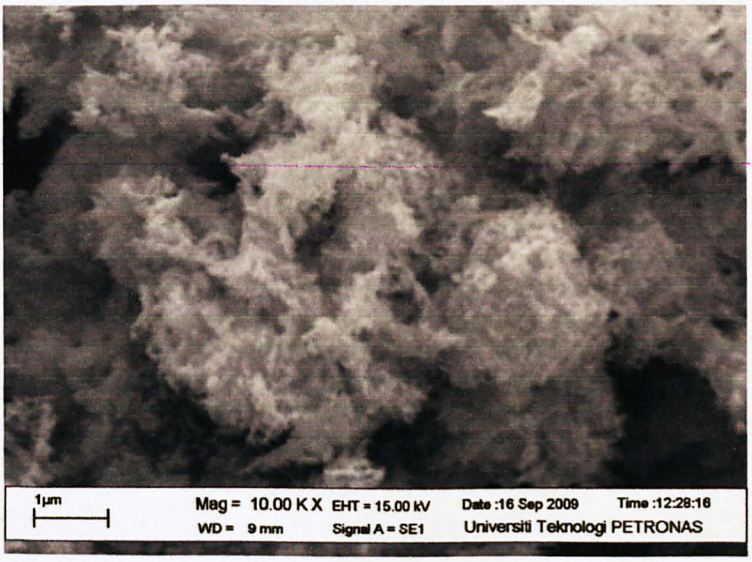


Figure 9: SEM at 10000K

The above figures are the results from SEM analysis that was prepared under 5000K and 10000K magnification. The figures show clearly the topography of $Cu/Zn/Al_2O_3$ fresh catalyst used in the project. Figures above show that the catalyst is not well dispersed and accumulated at specific spot. If compare to the industrial catalyst, industrial catalyst looks more solid, very well dispersed and the components look like very organized. Since that our catalyst is not well dispersed, this is affected our process rate of the reaction since the area of active sites on the catalyst is small.

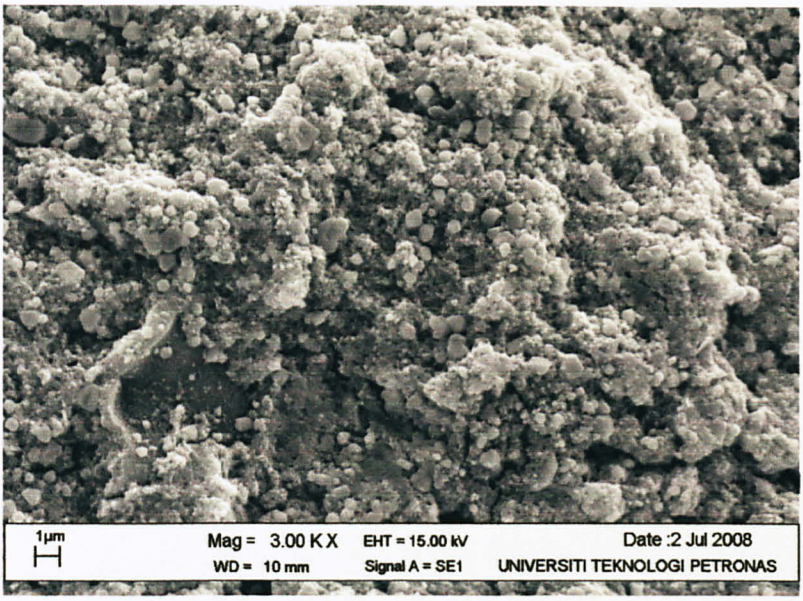


Figure 10: SEM result for Industrial Catalyst

4.2 X-RAY DIFFRACTION (XRD)

XRD analysis will determine the component on the catalyst and the structure of the catalyst. In this analysis, two types of catalyst is being tested which are fresh catalyst and spent catalyst. The XRD result is shown below.

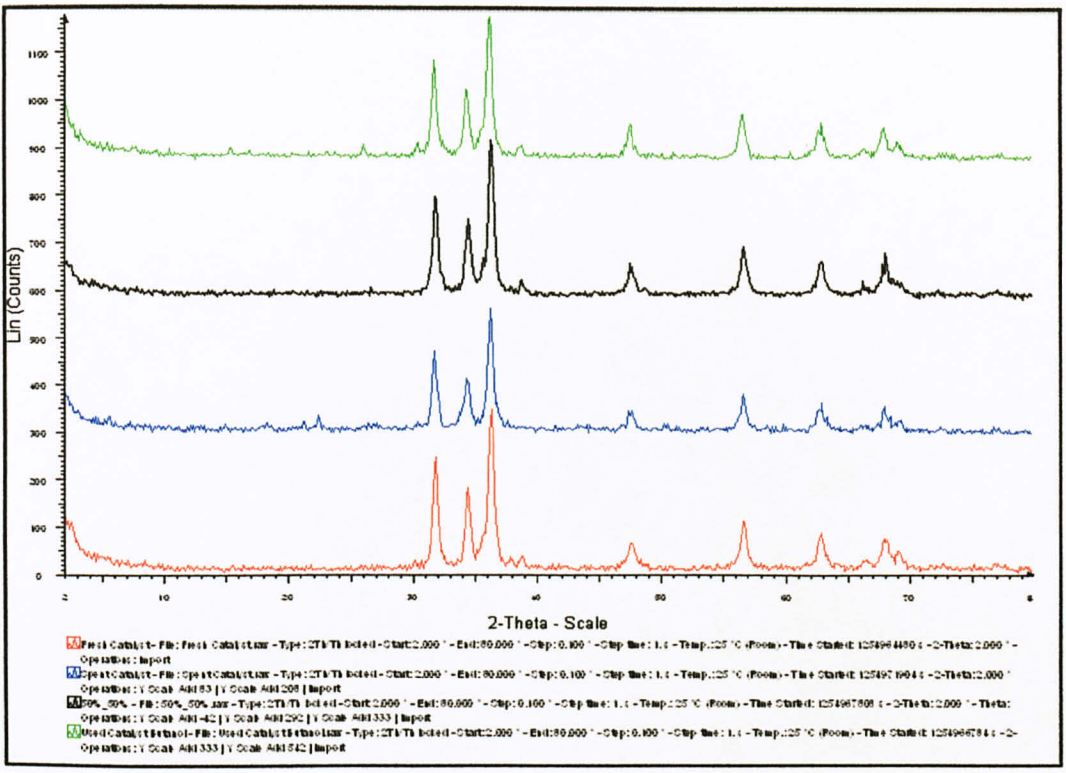


Figure 11: XRD Results

The results above show that the pattern or the peaks of the graph is almost similar. Red colored line indicate the fresh catalyst while green, black and blue colored line indicate the spent catalyst that has been used in methanol synthesis reaction via Parr High Pressure Reactor. Peaks that appear in XRD result shows that the catalyst is in crystal form. The same pattern of the peaks can be observed from the figure and this indicates that the catalyst has not be deactivated after the reaction of has took place. As a comparison to the industrial catalyst, the peak of both graph; graph for fresh catalyst used in this project and graph for industrial catalyst is quite the same. This indicates that the catalyst used in the project can function as well as industrial catalyst function. The different between the catalyst might be on the active sites area on the catalyst but not the function since that the structure on both catalyst is quite the same.

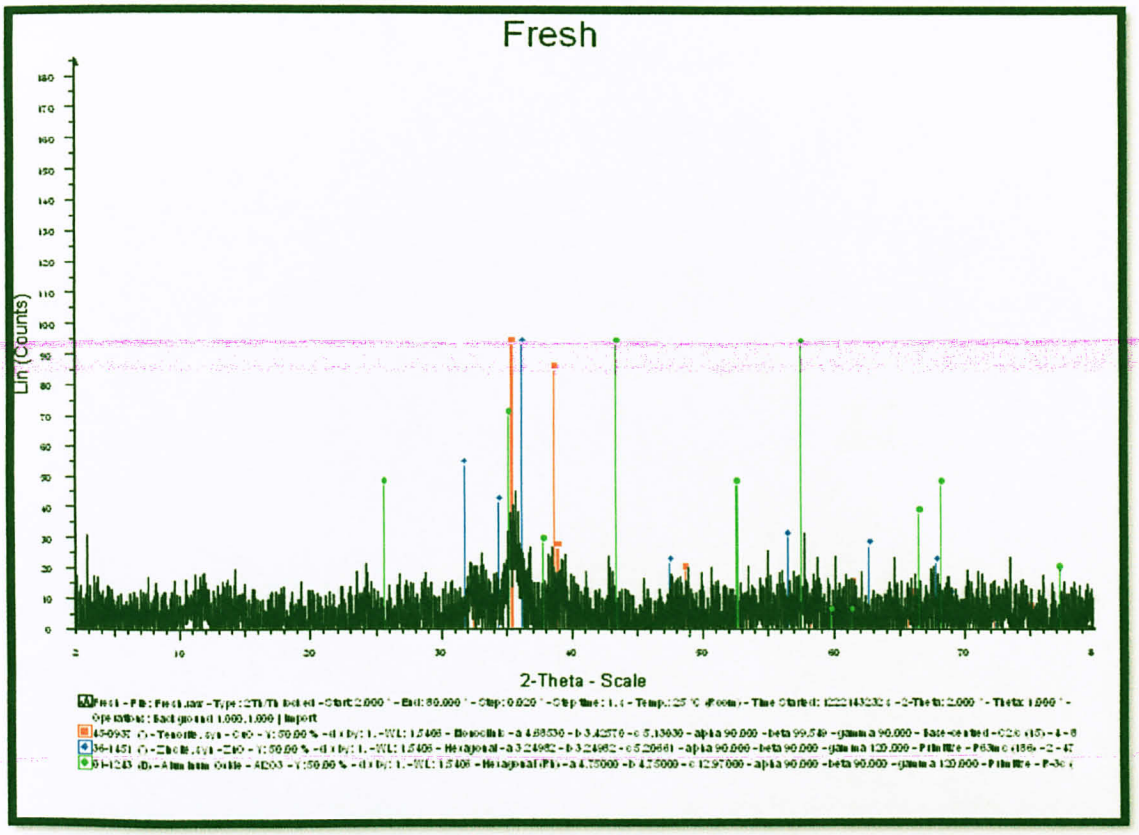


Figure 12: XRD results for Industrial Catalyst

4.3 Gas Chromatograph

In this project, the gas samples and liquid samples will be analyzed with gas chromatography. In this analysis, the results will give us the exact amount of methanol produced whether methanol is in the gas form or condensed to the liquid form.

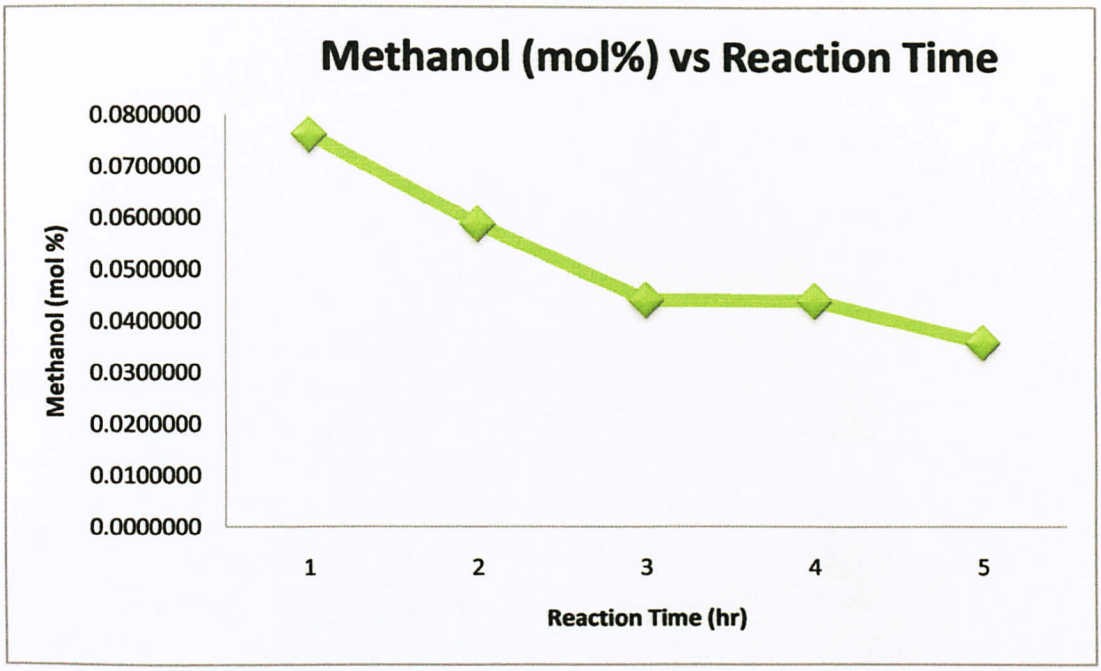


Figure 8: Amount of methanol in gas sample over time

The graph from Figure 8 is plotted using the gas sample data from Gas Chromatograph. From the graph, the amount of methanol in the gas samples is reducing in parallel time. At the first reaction hour, the amount of methanol is 0.08 % and 0.04% at last reaction hour. It is believed that the pressure inside the reactor force methanol gas produced to condense into liquid form at the surface of the solvent. Since that methanol and toluene cannot be mixed, two liquid layers will be seen. Besides that, other gases including reactant gases is not detected in gas samples. This shows that, toluene has a high tendency to absorb gas. This proves that the reaction is not happened at the surface of solvent where catalyst is floating, but the reaction happen inside the solvent.

Meanwhile in the liquid samples, there is no methanol detected. The only component detected inside liquid samples is ethanol. Thus, this result shows that the methanol is not favoured if toluene is used as catalytic solvent. There are two possibilities here. One will be the methanol is not been produced at all or two; the liquid samples is taken at the wrong place.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

As a conclusion, we can see that the methanol synthesis in the project is not favoured if toluene is use as a catalytic solvent. This is showed by the results in the previous chapter. Although the methanol is produced and detected only in the gas sample, its amount is too small and keeps reducing over time. The results give us some early prediction about the reaction conversion and product selectivity which are low conversion and low product selectivity.

There are a few improvements can be made to the project:

1. The study about the topic should be continued to get more knowledge about the project. The study should cover on reaction between methanol and toluene, optimum operating temperature in methanol synthesis and reaction of toluene with syngas.

2. The catalyst must be prepared more precisely. Every procedure must be followed. This is very important in producing more active catalyst thus affecting the rate of reaction in the process. The component inside catalyst must be well dispersed. This can be done by adding some sulfuric acid in the catalyst preparation process.

3. Some maintenance jobs should be done on the reactor. This is because during the experiment, the author has faced a few situations regarding gas leaking. There are some joint at the reactor is already loose and need to be tighten up. This has cause lost concentration of reactant gas inside the reactor.

4. The Tedlar bag that is used to take gas sample is not suitable for the reactor. Author need to modify the Tedlar bag fitting to match with the reactor gas outlet. This made the fitting is not properly connected and leads to lose of methanol due to leakage. A different Tedlar bag with a proper fitting should be used to enhance more accurate results.

REFERENCES

[1] Mayra.O and Leiviska.K , 2008, "*Modelling in Methanol Synthesis*", University of Oulu.

[2] Zhang.Y, Yang.R, Tsubaki.N, "*A new low-temperature methanol synthesis method: Mechanistic and kinetics study of catalytic process*" Catalysis Today 132 (2008) 93-100

[3] "Clean Fuel for the Future" by Methanol Australia, <www.methanol.com.au/_about-methanol.asp>

[4] Maier, *"5_1-Methanol-le-2008.ppt"* (2008)

[5] Terumitsu.K, and Watanabe.T, "*A theoretical study for methanol synthesis by CO_2 hydrogenation*", Catalyst Today 36 (1997) 39-44

[6] Tijm.P.J.A, Heydorn.E.C, Diamond.B.W, Street.B.T, Kornosky.R.M, *"Liquid Phase Methanol (LPMEOH^{TM}) Project: Operating Experience Update"* Presented Paper at 1999 Gasification Technologies Conference, October 17-20

[7] Hu.B, Yamaguchi.Y, Fujimoto.K, "*Low temperature methanol synthesis in alcohol solvent over cooper-based-catalyst*", Catalyst Communication (2009)

[8] Petersen.K.A, Nielsen.C.S, Dybkjaer.I, Perregaard.J, 2008, "*Large Scale Methanol Production from Natural Gas*", Haldor Topsoe

[9] "*Methanol Production*" retrieved on 8/2/2009 at 12.55 a.m, <www.methanol.org>

[10] Lattner.J.R," *Making a methanol product in a slurry bubble reactor*", U.S Patent (US 7459486 B2) (2008)

[11] Nemphos.P.S, Groten.W.A, Adams.J.R, "*Process for Production of Methanol*", U.S Patent (5886055) (1999)

[12] Eliasson.B, Kogelschatz.U, Xue.B, Zhou.L.M, "*Hydrogenation of carbon dioxide to methanol with a discharge-activated catalyst*" Ind.Eng.Chem.Res. (1998) 37,3350-3357

[13] Bao.J, Liu.Z, Zhang.Y, Tsubaki.N, "*Preparation of mesoporous Cu/ZnO catalyst and its application in low temperature methanol synthesis*" Catalyst Communication 9 (2008) 913-918

[14] Reubrocharoen P., Vitidsant T., Yoneyama Y., Tsubaki N., *"Development of a new low-temperature methanol synthesis process"* Catalysis Today 89 (2004) 447-454

[15] "Alternative & Advanced Fuel" by U.S Department of Energy, < http://www.afdc.energy.gov/afdc/fuels/methanol_basics.html >

[16] PARR Instrument Company, "HP/HT Pressure Reactor",2008

APPENDICES

ScienceLab.com
Chemicals & Laboratory Equipment

Health	2
Fire	3
Reactivity	0
Personal Protection	H

Material Safety Data Sheet
Toluene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Toluene

Catalog Codes: SLT2857, SLT3277

CAS#: 108-88-3

RTECS: XS5250000

TSCA: TSCA 8(b) inventory: Toluene

CI#: Not available.

Synonym: Toluol, Tolu-Sol; Methylbenzene; Methacide; Phenylmethane; Methylbenzol

Chemical Name: Toluene

Chemical Formula: C6-H5-CH3 or C7-H8

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Toluene	108-88-3	100

Toxicological Data on Ingredients: Toluene: ORAL (LD50): Acute: 636 mg/kg [Rat]. DERMAL (LD50): Acute: 14100 mg/kg [Rabbit]. VAPOR (LC50): Acute: 49000 mg/m 4 hours [Rat]. 440 ppm 24 hours [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Not available.
The substance may be toxic to blood, kidneys, the nervous system, liver, brain, central nervous system (CNS). Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.

Skin Contact:
In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek medical attention.

Ingestion:
Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 480°C (896°F)

Flash Points: CLOSED CUP: 4.4444°C (40°F). (Setaflash) OPEN CUP: 16°C (60.8°F).

Flammable Limits: LOWER: 1.1% UPPER: 7.1%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances:
Flammable in presence of open flames and sparks, of heat.
Non-flammable in presence of shocks.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available.
Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
Flammable liquid, insoluble in water.
SMALL FIRE: Use DRY chemical powder.
LARGE FIRE: Use water spray or fog.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards:
Toluene forms explosive reaction with 1,3-dichloro-5,5-dimethyl-2,4-imidazolididione; dinitrogen tetraoxide;

oncentrated nitric acid, sulfuric acid + nitric acid; N_2O_4; $AgClO_4$; BrF_3; Uranium hexafluoride; sulfur dichloride.
lso forms an explosive mixture with tetranitromethane.

Section 6: Accidental Release Measures

mall Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

arge Spill:
oxic flammable liquid, insoluble or very slightly soluble in water.
eep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth,
and or other non-combustible material. Do not get water inside container. Do not touch spilled material. Prevent
ntry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Be careful that
e product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

recautions:
eep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not
gest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient
entilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the
ontainer or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents.

torage:
tore in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly
losed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

Section 8: Exposure Controls/Personal Protection

ngineering Controls:
rovide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their
espective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the
ork-station location.

ersonal Protection:
plash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent.
loves.

ersonal Protection in Case of a Large Spill:
plash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be
sed to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist
EFORE handling this product.

xposure Limits:
WA: 200 STEL: 500 CEIL: 300 (ppm) from OSHA (PEL) [United States]
WA: 50 (ppm) from ACGIH (TLV) [United States] SKIN
WA: 100 STEL: 150 from NIOSH [United States]
WA: 375 STEL: 560 (mg/m3) from NIOSH [United States]
onsult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

hysical state and appearance: Liquid.

dor: Sweet, pungent, Benzene-like.

aste: Not available.

Molecular Weight: 92.14 g/mole

Color: Colorless.

pH (1% soln/water): Not applicable.

Boiling Point: 110.6°C (231.1°F)

Melting Point: -95°C (-139°F)

Critical Temperature: 318.6°C (605.5°F)

Specific Gravity: 0.8636 (Water = 1)

Vapor Pressure: 3.8 kPa (@ 25°C)

Vapor Density: 3.1 (Air = 1)

Volatility: Not available.

Odor Threshold: 1.6 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 2.7

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether, acetone.

Solubility:
Soluble in diethyl ether, acetone.
Practically insoluble in cold water.
Soluble in ethanol, benzene, chloroform, glacial acetic acid, carbon disulfide.
Solubility in water: 0.561 g/l @ 25 deg. C.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Heat, ignition sources (flames, sparks, static), incompatible materials

Incompatibility with various substances: Reactive with oxidizing agents.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Incompatible with strong oxidizers, silver perchlorate, sodium difluoride, Tetranitromethane, Uranium Hexafluoride.
Frozen Bromine Trifluoride reacts violently with Toluene at -80 deg. C.
Reacts chemically with nitrogen oxides, or halogens to form nitrotoluene, nitrobenzene, and nitrophenol and halogenated products, respectively.

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE.
Acute oral toxicity (LD50): 636 mg/kg [Rat].
Acute dermal toxicity (LD50): 14100 mg/kg [Rabbit].
Acute toxicity of the vapor (LC50): 440 24 hours [Mouse].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC.
May cause damage to the following organs: blood, kidneys, the nervous system, liver, brain, central nervous system (CNS).

Other Toxic Effects on Humans:
Hazardous in case of skin contact (irritant), of ingestion, of inhalation.
Slightly hazardous in case of skin contact (permeator).

Special Remarks on Toxicity to Animals:
Lowest Published Lethal Dose:
LDL [Human] - Route: Oral; Dose: 50 mg/kg
LCL [Rabbit] - Route: Inhalation; Dose: 55000 ppm/40min

Special Remarks on Chronic Effects on Humans:
Detected in maternal milk in human. Passes through the placental barrier in human. Embryotoxic and/or foetotoxic in animal. May cause adverse reproductive effects and birth defects (teratogenic). May affect genetic material (mutagenic)

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects:
Skin: Causes mild to moderate skin irritation. It can be absorbed to some extent through the skin.
Eyes: Cauess mild to moderate eye irritation with a burning sensation. Splash contact with eyes also causes conjunctivitis, blepharospasm, corneal edema, corneal abraisons. This usually resolves in 2 days.
Inhalation: Inhalation of vapor may cause respiratory tract irritation causing coughing and wheezing, and nasal discharge. Inhalation of high concentrations may affect behavior and cause central nervous system effects characterized by nausea, headache, dizziness, tremors, restlessness, lightheadedness, exhilaration, memory loss, insomnia, impaired reaction time, drowsiness, ataxia, hallucinations, somnolence, muscle contraction or spasticity, unconsciousness and coma. Inhalation of high concentration of vapor may also affect the cardiovascular system (rapid heart beat, heart palpitations, increased or decreased blood pressure, dysrhythmia,), respiration (acute pulmonary edema, respiratory depression, apnea, asphyxia), cause vision disturbances and dilated pupils, and cause loss of appetite.
Ingestion: Aspiration hazard. Aspiration of Toluene into the lungs may cause chemical pneumonitis. May cause irritation of the digestive tract with nausea, vomiting, pain. May have effects similar to that of acute inhalation.
Chronic Potential Health Effects:
Inhalation and Ingestion: Prolonged or repeated exposure via inhalation may cause central nervous system and cardiovascular symptoms similar to that of acute inhalation and ingestion as well liver damage/failure, kidney damage/failure (with hematuria, proteinuria, oliguria, renal tubular acidosis), brain damage, weight loss, blood (pigmented or nucleated red blood cells, changes in white blood cell count), bone marrow changes, electrolyte imbalances (Hypokalemia, Hypophostatemia), severe, muscle weakness and Rhabdomyolysis.
Skin: Repeated or prolonged skin contact may cause defatting dermatitis.

Section 12: Ecological Information

Ecotoxicity:
Ecotoxicity in water (LC50): 313 mg/l 48 hours [Daphnia (daphnia)]. 17 mg/l 24 hours [Fish (Blue Gill)]. 13 mg/l 96 hours [Fish (Blue Gill)]. 56 mg/l 24 hours [Fish (Fathead minnow)]. 34 mg/l 96 hours [Fish (Fathead minnow)]. 56.8 ppm any hours [Fish (Goldfish)].

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may

arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:
Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 3: Flammable liquid.

Identification: : Toluene UNNA: 1294 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Toluene
California prop. 65 (no significant risk level): Toluene: 7 mg/day (value)
California prop. 65 (acceptable daily intake level): Toluene: 7 mg/day (value)
California prop. 65: This product contains the following ingredients for which the State of California has found to cause birth defects which would require a warning under the statute: Toluene
Connecticut hazardous material survey.: Toluene
Illinois toxic substances disclosure to employee act: Toluene
Illinois chemical safety act: Toluene
New York release reporting list: Toluene
Rhode Island RTK hazardous substances: Toluene
Pennsylvania RTK: Toluene
Florida: Toluene
Minnesota: Toluene
Michigan critical material: Toluene
Massachusetts RTK: Toluene
Massachusetts spill list: Toluene
New Jersey: Toluene
New Jersey spill list: Toluene
Louisiana spill reporting: Toluene
California Director's List of Hazardous Substances.: Toluene
TSCA 8(b) inventory: Toluene
TSCA 8(d) H and S data reporting: Toluene: Effective date: 10/04/82; Sunset Date: 10/0/92
SARA 313 toxic chemical notification and release reporting: Toluene
CERCLA: Hazardous substances.: Toluene: 1000 lbs. (453.6 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).
EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F).
CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC):
R11- Highly flammable.
R20- Harmful by inhalation.
S16- Keep away from sources of ignition - No smoking.
S25- Avoid contact with eyes.
S29- Do not empty into drains.
S33- Take precautionary measures against static discharges.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves.
Lab coat.
Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate.
Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:30 PM

Last Updated: 11/06/2008 12:00 PM

ScienceLab.com
Chemicals & Laboratory Equipment

Health	2
Fire	3
Reactivity	0
Personal Protection	J

Material Safety Data Sheet
m-Xylene MSDS

Section 1: Chemical Product and Company Identification

Product Name: m-Xylene

Catalog Codes: SLX1066

CAS#: 108-38-3

RTECS: ZE2275000

TSCA: TSCA 8(b) inventory: m-Xylene

CI#: Not applicable.

Synonym: m-Methyltoluene

Chemical Name: 1,3-Dimethylbenzene

Chemical Formula: C6H4(CH3)2

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
{m-}Xylene	108-38-3	100

Toxicological Data on Ingredients: m-Xylene: ORAL (LD50): Acute: 5000 mg/kg [Rat.]. DERMAL (LD50): Acute: 14100 mg/kg [Rabbit.].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of skin contact (irritant), of eye contact (irritant). Slightly hazardous in case of skin contact (permeator), of ingestion, of inhalation. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:
Hazardous in case of skin contact (irritant), of eye contact (irritant).
Slightly hazardous in case of skin contact (permeator), of ingestion, of inhalation.
CARCINOGENIC EFFECTS: Not available.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Not available.
The substance is toxic to blood, kidneys, the nervous system, liver.
Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact: Check for and remove any contact lenses. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation: Not available.

Ingestion:
Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 527°C (980.6°F)

Flash Points: CLOSED CUP: 25°C (77°F). OPEN CUP: 28.9°C (84°F) (Cleveland).

Flammable Limits: LOWER: 1.1% UPPER: 7%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances: Highly flammable in presence of open flames and sparks, of heat.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available.
Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
Flammable liquid, insoluble in water.
SMALL FIRE: Use DRY chemical powder.
LARGE FIRE: Use water spray or fog. Cool containing vessels with water jet in order to prevent pressure build-up, autoignition or explosion.

Special Remarks on Fire Hazards:
Explosive in the form of vapor when exposed to heat or flame. Vapor may travel considerable distance to source of ignition and flash back. When heated to decomposition it emits acrid smoke and irritating fumes.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Flammable liquid, insoluble in water.

Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Eliminate all ignition sources. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/spray. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes Keep away from incompatibles such as oxidizing agents.

Storage:
Flammable materials should be stored in a separate safety storage cabinet or room. Keep away from heat. Keep away from sources of ignition. Keep container tightly closed. Keep in a cool, well-ventilated place. Ground all equipment containing material. A refrigerated room would be preferable for materials with a flash point lower than 37.8°C (100°F).

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection: Splash goggles. Lab coat. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Boots. Gloves. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 100 STEL: 150 (ppm) from ACGIH (TLV)
TWA: 434 STEL: 651 (mg/m3) from ACGIHConsult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid. (Liquid.)

Odor: Not available.

Taste: Not available.

Molecular Weight: 106.17 g/mole

Color: Colorless.

pH (1% soln/water): Not applicable.

Boiling Point: 139.3°C (282.7°F)

Melting Point: -47.87°C (-54.2°F)

Critical Temperature: Not available.

Specific Gravity: 0.86 (Water = 1)

Vapor Pressure: 6 mm of Hg (@ 20°C)

Vapor Density: 3.7 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.62 ppm

Water/Oil Dist. Coeff.: Not available.

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, methanol, diethyl ether.

Solubility:
Easily soluble in methanol, diethyl ether.
Insoluble in cold water, hot water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Reactive with oxidizing agents.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Eye contact.

Toxicity to Animals:
Acute oral toxicity (LD50): 5000 mg/kg [Rat.].
Acute dermal toxicity (LD50): 14100 mg/kg [Rabbit.].

Chronic Effects on Humans: The substance is toxic to blood, kidneys, the nervous system, liver.

Other Toxic Effects on Humans:
Very hazardous in case of skin contact (irritant).
Slightly hazardous in case of skin contact (permeator), of ingestion, of inhalation.

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans:
0347 Animal: embryotoxic, foetotoxic, passes through the placental barrier.
0900 Detected in maternal milk in human.
Narcotic effect; may cause nervous system disturbances.

Special Remarks on other Toxic Effects on Humans: Material is irritating to mucous membranes and upper respiratory tract.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: Class 3: Flammable liquid.

Identification: : Xylene : UN1307 PG: III

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
Pennsylvania RTK: m-Xylene
Massachusetts RTK: m-Xylene
TSCA 8(b) inventory: m-Xylene
SARA 313 toxic chemical notification and release reporting: m-Xylene
CERCLA: Hazardous substances.: m-Xylene

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):
CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F).
CLASS D-2B: Material causing other toxic effects (TOXIC).

DSCL (EEC):
R10- Flammable.
R38- Irritating to skin.
R41- Risk of serious damage to eyes.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: j

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves.
Lab coat.
Wear appropriate respirator when ventilation is inadequate.
Splash goggles.

Section 16: Other Information

References:
-Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987.
-Material safety data sheet emitted by: la Commission de la SantÃ© et de la SÃ©curitÃ© du Travail du QuÃ©bec.
-SAX, N.I. Dangerous Properties of Indutrial Materials. Toronto, Van Nostrand Reinold, 6e ed. 1984.
-The Sigma-Aldrich Library of Chemical Safety Data, Edition II.
-Guide de la loi et du rÃ¨glement sur le transport des marchandises dangeureuses au canada. Centre de conformitÃ© internatinal LtÃ©e. 1986.

Other Special Considerations: Not available.

Created: 10/10/2005 08:33 PM

Last Updated: 11/06/2008 12:00 PM